

PATENT ABSTRACTS OF JAPAN

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(54) DISSOLUTION OF RAW MATERIAL OF SILICA TYPE FLOCCULATING SOLUTION,
METHOD FOR PRODUCING SILICA TYPE FLOCCULATING SOLUTION AND APPARATUS
THEREFOR

(57)Abstract:

PURPOSE: To obtain a silica type flocculating soln. high in the content of monomer silica and having high stability by charging a silica type flocculating soln. raw material having specific Ca basicity of a dissolution equilibrium amt. capable of being dissolved by a dilute sulfuric acid into a dissolving tank wherein the dilute sulfuric acid with predetermined concn. is prepared.

CONSTITUTION: A fixed amt. of water is supplied to a dissolving tank and sulfuric acid is poured in the tank while the water in the tank is stirred to prepare dilute sulfuric acid with predetermined concn. A silica type flocculating soln. raw material having a specific Ca basicity, for example, Ca basicity of about 1.5 is charged to the dissolving tank and held until it reaches dissolution equilibrium. Next, if necessary, it is judged whether the pH in the dissolving tank exceeds 2.5 and, when the pH is below 2.5, the prepared soln. is held for several min until the pH exceeds 2.5 and, when the pH is 2.5 or more, it is judged whether filtering is performed and, when no filtering is performed, the prepared soln. is used as a silica type flocculating soln. as it is and, in the case of filtering, a filtrate is used as the silica type flocculating soln.

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DETAILED DESCRIPTION

[Detailed Description of the Invention]

[0001]

[Industrial Application]The dissolved solid, the emulsion substance, the dispersing agent, oil which contain this invention underwater, A dissolving method of the silica system condensation liquid raw material used as the raw material for manufacturing the silica system condensation liquid used for adsorption of a suspended solid etc., condensation, and exclusion, It is related with the dissolving method of the silica system condensation liquid raw material for obtaining detailed silica system condensation liquid with a high content ratio of monomer silica, the manufacturing method of silica system condensation liquid, and the manufacturing installation of the silica system condensation liquid in which a compact design is possible about the manufacturing method and device of silica system condensation liquid.

[0002]

[Background of the Invention]Conventionally, aluminum system condensation liquid, Fe system condensation liquid, and polymers system condensation liquid are mainly used for extraction of the dissolved solid (ion) in sewage or various waste water, organic and inorganic particles, a suspended solid, etc., etc., or condensation and removal.

[0003]Since the function which this conventional aluminum system and Fe system condensation liquid adsorb an organic chemistry substance, oil, an oil emulsion, a surface-active agent, etc., or it condenses is very weak, it faces actually using it, Precipitation of a lot of aluminium hydroxide and iron hydroxide is made to generate using a lot of aluminum systems and Fe system condensation liquid (also adding alkali further), Oil etc. are physically hooked on those settlings, and also a polymer coagulant is prescribed for the patient, flocks shape is enlarged, and floatation or the way sedimentation separates flocks with water is taken.

[0004]Even if it performs this complicated processing, it is difficult to obtain a satisfactory result, and also a lot of sludge resulting from the flocculating agent prescribed for the patient

occurs, and there is a problem which must also pay the cleanup cost collectively.

[0005]In order to solve this problem, silica system condensation liquid is proposed in the Japanese-Patent-Application-No. No. 296371 [one to] specification. The profits that the sludge of this silica system condensation liquid which adsorbs effectively those organic chemistry substances, oil, an oil emulsion, a surface-active agent, etc., condenses them, and is generated also decreases in a degree very much are obtained.

[0006]Although obtained by conventional silica system condensation liquid dissolving a silica system condensation liquid raw material in neutral acid or the reductic acid, and a concrete target at dilute sulfuric acid, When dissolving a silica system condensation liquid raw material, after dissolving a silica system condensation liquid raw material in water, by the method of adding dilute sulfuric acid, it turned out that a silica system condensation liquid raw material becomes sol gel, and there is a fault which lacks in stability few in the rate of monomer silica. Although the statement of the purport that a silica system condensation liquid raw material is added to dilute sulfuric acid is accepted in conventional technology, recognition of the purport that an addition order influences the rate of monomer silica greatly does not exist at all.

[0007]Although the silica system condensation liquid which dissolved double the amount of the raw material from the efficiency of transportation, etc. was called for, it turned out that the monomer silica needed depending on a manufacturing method decreases. Namely, if a silica system condensation liquid raw material is dissolved in 1 l. of dilute sulfuric acid of 1N by the dissolution equivalent, the silica system condensation liquid of 1N will be obtained, but. If the dissolution equivalent part (twice at time of 1N as many quantity as this) dissolution of the silica system condensation liquid raw material is carried out, to 1 l. of dilute sulfuric acid of 2N, noting that I will obtain the silica system condensation liquid of 2N like this, In the place where pH rises selectively by addition of a silica system condensation liquid raw material, polymerization-ization of silica took place and it turned out in the low place of pH that anhydrous silica is generated and the monomer silica needed decreases.

[0008]furthermore -- needing at least 2 liquid, when using conventional aluminum system condensation liquid, Fe system condensation liquid, and polymers system condensation liquid -- so much -- also coming out -- the number of devices increased and miniaturization of the device was made difficult. In the art of Japanese Patent Application No. No. 296371 [one to], the concrete manufacturing method and device of silica system condensation liquid were not clear, and development of a manufacturing method and a device applicable to a actual device was desired.

[0009]

[Problem(s) to be Solved by the Invention]Then, there is a technical problem of this invention in providing the dissolving method of the silica system condensation liquid raw material in which extremely stable silica system condensation liquid with many content ratios of monomer silica

is obtained [1st].

[0010]The 2nd technical problem has a transportation cost in providing the dissolving method of the silica system condensation liquid raw material which it is low, and economic effectuation is high, and can dissolve a several times the amount raw material.

[0011]There is the 3rd technical problem in providing the manufacturing method of silica system condensation liquid applicable to a actual device.

[0012]There is the 4th technical problem in providing the manufacturing installation of compact silica system condensation liquid.

[0013]

[Means for Solving the Problem]this invention person used to result in this invention, as a result of repeating examination wholeheartedly that an aforementioned problem should be solved.

[0014]Namely, the 1st of a dissolving method of a silica system condensation liquid raw material concerning this invention, Supplying a constant rate of water to a dissolver, and agitating water in this dissolver, when dissolving a silica system condensation liquid raw material with dilute sulfuric acid and manufacturing silica system condensation liquid. After pouring in sulfuric acid and creating dilute sulfuric acid of prescribed concentration, the amount of solution equilibriums which can dissolve a silica system condensation liquid raw material which has the specific degree of Ca salt group with said dilute sulfuric acid is supplied in this dissolver.

[0015]The 2nd of a dissolving method of a silica system condensation liquid raw material concerning this invention, In a dissolving method of a silica system condensation liquid raw material in which a content ratio of monomer silica is high, and high concentration silica system condensation liquid is obtained, Silica system condensation liquid is obtained by the 1st method of the above, subsequently sulfuric acid is added by the same equivalent as said dilute sulfuric acid, the amount of solution equilibriums which can dissolve said silica system condensation liquid raw material with this dilute sulfuric acid again is supplied, and this operation is repeated as occasion demands.

[0016]A manufacturing method of silica system condensation liquid concerning this invention, In a manufacturing method of silica system condensation liquid which dissolves a silica system condensation liquid raw material which has the specific degree of Ca salt group with dilute sulfuric acid, and manufactures silica system condensation liquid, After pouring in sulfuric acid and creating dilute sulfuric acid of prescribed concentration, supplying a constant rate of water to a dissolver, and agitating water in this dissolver, After holding until it throws in a silica system condensation liquid raw material and reaches subsequently to a solution equilibrium in this dissolver, Next, it judges whether buys and there is that pH in this dissolver is over 2.5 as occasion demands, In holding for several minutes, judging whether it next filters when pH is

2.5 or more on the other hand, considering it as silica system condensation liquid as it is in not filtering and filtering until pH exceeds 2.5, when this pH is less than 2.5, let filtrate be silica system condensation liquid.

[0017]This invention is characterized by a manufacturing installation of silica system condensation liquid comprising the following, in order to pour sulfuric acid into a dissolver to which water was supplied from a dissolver, a water supplying means which supplies a constant rate of water to this dissolver, an agitating means which agitates liquid in this dissolver, and said water supplying means.

Sulfuric acid injection means.

A means to supply a silica system condensation liquid raw material to this dissolver.

A condensation fluid tank for receiving silica system condensation liquid, when pH in this dissolver is 2.5 or more and it does not filter a solution in this dissolver.

[0018]This invention is characterized by a manufacturing installation of silica system condensation liquid comprising the following again, in order to pour sulfuric acid into a dissolver to which water was supplied from a dissolver, a water supplying means which supplies a constant rate of water to this dissolver, an agitating means which agitates liquid in this dissolver, and said water supplying means.

Sulfuric acid injection means.

A means to supply a silica system condensation liquid raw material to this dissolver.

A condensation fluid tank for receiving silica system condensation liquid which pH in this dissolver is 2.5 or more, and is this filtrate when filtering a solution in this dissolver.

[0019]Hereafter, this invention is explained in detail.

[0020]The 1st feature of a dissolving method of a silica system condensation liquid raw material, After pouring in sulfuric acid and creating dilute sulfuric acid of prescribed concentration, supplying a constant rate of water to a dissolver, and agitating liquid in this dissolver, it is supplying the amount of solution equilibriums which can dissolve a silica system condensation liquid raw material which has the specific degree of Ca salt group with said dilute sulfuric acid in this dissolver. Namely, when it was essence, and throwing in a silica system condensation liquid raw material adds sulfuric acid after [this] creating solution of a silica system condensation liquid raw material conversely after creating a dilution-water solution of dilute sulfuric acid. A silica system condensation liquid raw material becomes sol gel, a rate of monomer silica decreases, and the purpose of this invention cannot be attained.

[0021]The 2nd feature of a dissolving method of a silica system condensation liquid raw material, After pouring in sulfuric acid and creating dilute sulfuric acid of prescribed concentration, supplying a constant rate of water to a dissolver, and agitating liquid in this

dissolver, Supply the amount of solution equilibriums which can dissolve a silica system condensation liquid raw material which has the specific degree of Ca salt group with said dilute sulfuric acid in this dissolver, and silica system condensation liquid is obtained, Subsequently, it is the method of adding sulfuric acid by the same equivalent as said dilute sulfuric acid, and also supplying the amount of solution equilibriums which can dissolve said silica system condensation liquid raw material with this dilute sulfuric acid, and obtaining high-concentration silica system condensation liquid.

[0022]Supplying water to a dissolver and agitating liquid in this dissolver, for example, if how to obtain this high-concentration silica system condensation liquid is explained concretely. After pouring in conc sulfuric acid and creating 1 l. of dilute sulfuric acid of 1N concentration, Supply the amount of solution equilibriums of 42.5 g which can dissolve a silica system condensation liquid raw material which has the degree 1.15 of Ca salt group with dilute sulfuric acid of said 1N concentration in this dissolver, and silica system condensation liquid is obtained, Subsequently, sulfuric acid can be added by the same equivalent as said dilute sulfuric acid ($\text{H}_2\text{SO}_4=49\text{g}$), and also the amount of solution equilibriums of 42.5 g which can dissolve said silica system condensation liquid raw material with this dilute sulfuric acid can be supplied, and what is called high concentration silica system condensation liquid of 2N can be obtained. It is obtained by repeating this operation similarly about 3N, 4N, and 5N.

[0023]On the other hand, supplying water to a dissolver and agitating liquid in this dissolver. If the amount of solution equilibriums of 85 g which can dissolve a silica system condensation liquid raw material which has the degree 1.15 of Ca salt group with dilute sulfuric acid of said 2N concentration is supplied in this dissolver and silica system condensation liquid is obtained after pouring in conc sulfuric acid and creating 1 l. of dilute sulfuric acid of 2N concentration, Although it is thought that what is called high concentration silica system condensation liquid of 2N can be obtained seemingly after all, Thus, as for obtained silica system condensation liquid, polymerization-ization of silica takes place in a place where pH rises selectively by addition of a silica system condensation liquid raw material, and anhydrous silica is generated in a low place of pH, There is a problem that monomer silica needed will decrease and the purpose of this invention cannot be attained.

[0024]

[Example]Next, the example of the manufacturing method of the silica system condensation liquid concerning this invention is described based on a drawing.

[0025]Drawing 1 is a manufacture system flow figure of silica system condensation liquid.

[0026]A switch is turned on, a system is started and a constant rate of water is first supplied to a dissolver. This water is not exceptionally limited, although tap water is usually used.

However, the water which contains not less than several PPM for metal ions, such as Fe ion and Al ion, starts silica and an interaction, and is not preferred.

[0027]Next, if the quantity of water becomes fixed, the water in a dissolver will be agitated.

[0028]Next, sulfuric acid is poured in and dilute sulfuric acid of prescribed concentration is created. In the meaning which prevents gelling of monomer silica, as for dilute-sulfuric-acid concentration, less than 2N is preferred, and it is less than 1N more preferably. In this example, it is considered as 1N dilute sulfuric acid.

[0029]As for the sulfuric acid to pour in, when concentrated sulfuric acid is used and acceptance equipment etc. are taken into consideration, accepting by concentration 64% is preferred. It can also be used when existing has concentrated-sulfuric-acid equipment (Fire Service Law full equipment) 98%.

[0030]Next, it holds for several minutes (it is neglected under churning). It is for obtaining 1N uniform dilute sulfuric acid. There should just be about 3 minutes of retention time.

[0031]Next, a silica system condensation liquid raw material is thrown in in a dissolver. As used in this invention, the "amount of solution equilibriums" of a silica system condensation liquid raw material means the quantity of the silica system condensation liquid raw material which can dissolve in the given amount of sulfuric acid. The quantity of the silica system condensation liquid raw material of the degree 1.15 of Ca salt group which can dissolve with 1 l. of 1N dilute sulfuric acid in this example is 42.5g.

[0032]Next, it holds until it reaches a solution equilibrium. Although about 5 minutes may be sufficient as retention time, by the time it reaches a solution equilibrium more certainly, for 15 minutes is preferred.

[0033]Next, it judges whether buys and there is that pH in a dissolver is over 2.5 as occasion demands. If the reaction of dilute sulfuric acid and a raw material advances, pH in a dissolver will rise. And it holds for several minutes until pH exceeds 2.5, since the reaction is not yet advancing to a solution equilibrium when pH is less than 2.5. By this maintenance, a reaction advances and pH rises. If a reaction is made to continue, in order that pH may converge, if the convergence relation of the reaction time and pH is held, this pH judgment is unnecessary daringly. When attaining low cost-ization in an equipment surface, it is preferred to take the method which does not make this pH judgment.

[0034]On the other hand, since it can judge that the reaction advanced and the solution equilibrium was reached when pH is 2.5 or more, it is judged whether next it filters.

[0035]And it accepts in a condensation fluid tank, including sludge, when not filtering. The silica system condensation liquid of this invention can be obtained by this.

[0036]In filtering, a filter separates into filtrate and sludge, and filtrate is received in a condensation fluid tank. The silica system condensation liquid of this invention can be obtained by this.

[0037]As for the last pH within the dissolver in the case of filtering, it is also preferred to increase and adjust the quantity of a silica system condensation liquid raw material so that it

may be set to pH**3.0.

[0038]Sludge is CaSO_4 and $2\text{H}_2\text{O}$ (gypsum fibrosum), and accepts this for example, in a sludge pit.

[0039]When the silica system condensation liquid of this invention in a condensation fluid tank is used, it decreases and it is detected with a level sensor, it returns to a start and the above-mentioned method is repeated again.

[0040]As for the above-mentioned system, automating is preferred, and it clarifies concrete interlocking relationship in the following examples of a device.

[0041]The silica system condensation liquid raw materials used for the manufacturing method of the silica system condensation liquid of this invention are the composite crystal mineral and amorphous mineral of silica.

For example, natural cristobalite, blast furnace slag, etc. are used.

Although silica system condensation liquid raw materials by which reforming treatment was carried out, such as oxidation treatment, i.e., the silica system condensation liquid raw material from which the non-sulfur oxide ingredient of the composite crystal mineral and the amorphous mineral of silica was removed, are specifically preferred, the silica system condensation liquid raw material indicated to Japanese Patent Application No. No. 296371 [one to] can also be used.

[0042]Next, the example of the manufacturing installation of the silica system condensation liquid concerning this invention is described based on drawing 2.

[0043]In drawing 2, 1 is a sulfuric acid injection means, for example, comprises the sulfuric acid tank 101 received as sulfuric acid 64%, the sulfuric acid pump 102, the pipette-like sulfuric acid fixed-quantity container 103, and the sulfuric acid injection valve 104. A corrosion-resistant pump is used for the sulfuric acid pump 102. The pipette-like sulfuric acid fixed-quantity container 103 makes the sulfuric acid sent from the sulfuric acid pump 102 overflow from the overflow pipe 105 in which it was provided by the small tube part of the sulfuric acid fixed-quantity container 103, and is constituted so that a fixed quantity is possible.

[0044]2 is a dissolver and is formed with the product made of resin, or an anticorrosion metal. 201 is an agitator for agitating the liquid in the dissolver 2, and 3 is a tap water injection valve which constitutes a tap water injection means.

[0045]In the dissolver 2, the high-level sensor 202 and the low level sensor 203 are formed. The high-level sensor 202 is being interlocked with the sulfuric acid pump 102, the sulfuric acid injection valve 104, and the agitator 201 respectively, and the low level sensor 203 is being interlocked with the sulfuric acid injection valve 104, the agitator 201, and the tap water injection valve 3 respectively.

[0046]204 is a pH meter formed as occasion demands in the dissolver 2, and is taken as the index which that pH is over 2.5 judges whether buys and there is. However, since a pH value

is converged by the reaction time of sulfuric acid and a raw material as mentioned above, a sequence can also be constructed by a timer.

[0047]4 is a means to supply the silica system condensation liquid raw material 401 to the dissolver 2, and is constituted by the feeding feeder 403 formed when carrying out automatic feed to the hopper 402. To not carry out automatic feed, the feeding feeder 403 is unnecessary. In the hopper 402, it may have an air feeding means for oxidizing the silica system condensation liquid raw material 401 within a hopper.

[0048]5 is a condensation fluid tank for receiving silica system condensation liquid.

[0049]pH is 2.5 or more, and the solution obtained within the dissolver 2 is directly sent to the condensation fluid tank 5 with the liquid-sending pump 501, when not filtering. 502 is an agitator and 503 is a valve.

[0050]On the other hand, pH is 2.5 or more, and the solution obtained within the dissolver 2 is sent to the filter 6 with the liquid-sending pump 501, when filtering a solution. In the filter 6, it separates into filtrate and sludge and filtrate is sent to the condensation fluid tank 5. Sludge is stored by the sludge pit 601.

[0051]Various waste water treatment is presented with the silica system condensation liquid in the condensation fluid tank 5 with the treated water pump 7. The treated water pump 7 stops by detection of the low level sensor 504.

[0052]When pH is 2.5 or more and the solution obtained within the above-mentioned dissolver 2 does not filter, various waste water treatment can also be directly presented via the line 505 with the liquid-sending pump 501.

[0053]Next, although the main sequences preferably adopted as the dissolving method of this invention and the manufacturing method of silica system condensation liquid, and a device are explained, it is not necessarily limited.

[0054]Sulfuric acid is received in the sulfuric acid injection means 1 64%, and specified quantity measuring is carried out at the sulfuric acid fixed-quantity container 103. moreover -- opening the tap water injection valve 3 -- the inside of the dissolver 2 -- water -- ** -- a fixed quantity is filled.

[0055]The agitator 210 is started in this state. Subsequently, the sulfuric acid injection valve 104 is opened and the specified quantity is poured in.

[0056]For [time lag] 5 minutes is provided, and if the feeding feeder 403 is started and specified quantity supply is carried out, it will stop.

[0057]For [time lag] 15 minutes is provided, the valve 503 is opened, and the liquid-sending pump 501 is started. Operation of this liquid-sending pump 501 is interlocked with the filter 6 (however, when it needs filtration). Therefore, the filter 6 is also started simultaneously with starting of the liquid-sending pump 501.

[0058]By detection of the low level sensor 203, the liquid-sending pump 501 is suspended, the

valve 503 is closed and operation of the filter 6 is suspended.

[0059]Filtrate is received in the condensation fluid tank 5, and the agitator 502 is run continuously at this time.

[0060]If the treated water pump 7 operates, the oil level in the condensation fluid tank 5 falls and the low level sensor 504 detects, the treated water pump 7 will stop, and the tap water injection valve 3 will open, and tap water will be supplied to the dissolver 2. The tap water injection valve 3 is closed by detection of the high-level sensor 202.

[0061]By detection of the low level sensor 203, the tap water injection valve 3 is opened and tap water can be supplied to the dissolver 2.

[0062]

[Example(s) of Experiment]

It experimented using the device shown in example of experiment 1 drawing 2. First, the tap water injection valve 3 was opened to the 150-l. dissolver 2, and 100 l. of tap water was supplied to it.

[0063]Agitating the liquid in this dissolver 2 with the agitator 201, the sulfuric acid injection valve 104 is opened, 4.34 l. of sulfuric acid is poured in 64%, and it was made to become dilute sulfuric acid of 1N concentration.

[0064]Subsequently, the silica system condensation liquid raw material which has the degree 1.15 of Ca salt group was thrown in for 4.25 kg in this dissolver 2.

[0065]Churning was continued and held for 15 minutes and silica system condensation liquid was obtained.

[0066]The viscosity of this silica system condensation liquid was measured as an index, and the polymerization gelling state was investigated. It settled and sol gelling stages of progress were investigated.

[0067]The viscosity meter performed measurement of viscosity.

[0068]A measurement result is shown in Table 1.

[0069]It experimented using the device shown in drawing 2 like the example 1 of example of comparative experiments 1 experiment. First, the tap water injection valve 3 was opened to the 150-l. dissolver 2, and 100 l. of tap water was supplied to it.

[0070]Subsequently, the silica system condensation liquid raw material which has the degree 1.15 of Ca salt group was thrown in for 4.25 kg in the dissolver 2, agitating the liquid in the dissolver 2 with the agitator 201.

[0071]Subsequently, the sulfuric acid injection valve 104 is opened, 4.34 l. of sulfuric acid is poured in 64%, and it was made to become dilute sulfuric acid of 1N concentration.

[0072]Churning was continued and held for 15 minutes and silica system condensation liquid was obtained.

[0073]The viscosity of this silica system condensation liquid was measured as an index, and

the polymerization and the gelling state were investigated.

[0074]Measurement of viscosity was made to be the same as that of the example 1 of an experiment.

[0075]A measurement result is shown in Table 1.

[0076]

[Table 1]

	シリカ系凝集液の粘度	ゲル化の進行
実施例 1	1.4 センチポイズ	168 時間後も安定
比較実施例 1	2.3 センチポイズ	24 時間後ゾルゲル化→48 時間後ゲル化

It turns out that the polymerization of silica and gelling are not following the silica system condensation liquid of this invention so that clearly from Table 1. Although sol gelling was accepted 24 hours afterward and gelled 48 hours afterward in the example 1 of comparative experiments, the example 1 of an experiment was stable.

[0077]It experimented using the device shown in example of experiment 2 drawing 2. First, the tap water injection valve 3 was opened to the 150-l. dissolver 2, and 100 l. of tap water was supplied to it.

[0078]Agitating the water in this dissolver 2 with the agitator 201, the sulfuric acid injection valve 104 is opened, 4.34 l. of sulfuric acid is poured in 64%, and it was made to become dilute sulfuric acid of 1N concentration.

[0079]Subsequently, the silica system condensation liquid raw material which has the degree 1.15 of Ca salt group was thrown in for 4.25 kg in this dissolver 2. Stirring was continued for 15 minutes and the silica system condensation liquid of 1N dilute-sulfuric-acid dissolution was obtained.

[0080]4.34 l. was added for sulfuric acid 64%, and the silica system condensation liquid raw material which has the degree 1.15 of Ca salt group in this was thrown in for 4.25 kg.

[0081]Churning was continued and held for 15 minutes and high-concentration silica system condensation liquid was obtained.

[0082]The viscosity of this silica system condensation liquid was measured as an index, the polymerization and the gelling state were investigated and settled, and the stages of progress of sol gelling were investigated.

[0083]Measurement of viscosity was made to be the same as that of the example 1 of an experiment.

[0084]A measurement result is shown in Table 2.

[0085]It experimented using the device shown in drawing 2 like the example 2 of example of comparative experiments 2 experiment. First, the tap water injection valve 3 was opened to the 150-l. dissolver 2, and 100 l. of tap water was supplied to it.

[0086]Agitating the liquid in this dissolver 2 with the agitator 201, the sulfuric acid injection valve 104 is opened, 8.68 l. of sulfuric acid is poured in 64%, and it was made to become dilute sulfuric acid of 2N concentration.

[0087]Subsequently, the silica system condensation liquid raw material which has the degree 1.15 of Ca salt group was thrown in for 8.5 kg in this dissolver 2.

[0088]Churning was continued and held for 15 minutes and high-concentration silica system condensation liquid was obtained.

[0089]The viscosity of this silica system condensation liquid was measured as an index, the polymerization and the gelling state were investigated and settled, and the stages of progress of sol gelling were investigated.

[0090]Measurement of viscosity was made to be the same as that of the example 1 of an experiment.

[0091]A measurement result is shown in Table 2.

[0092]

[Table 2]

	シリカ系凝集液の粘度	ゲル化の進行
実施例 1	1.3 センチポイズ	168 時間後も安定
比較実施例 1	2.1 センチポイズ	24 時間後ゾルゲル化→48 時間後ゲル化

The silica system condensation liquid of this invention is understood that the content of monomer silica is clearly high in spite of being high concentration, and the stability of monomer silica is high so that clearly from Table 2.

[0093]That there are few monomer silica contents of comparative silica system condensation liquid, and the stability of monomer silica is missing, In the place where pH rises selectively by addition of a silica system condensation liquid raw material, polymerization-ization of silica takes place and it is thought in the low place of pH that it originates in anhydrous silica being generated. If the polymerization of silica and sol gelling are accepted, gelling will advance quickly and it will become a jelly state solid.

[0094]

[Effect of the Invention]according to this invention, the content ratio of monomer silica highly so that clearly from the above explanation. [1st] The dissolving method of the silica system condensation liquid raw material in which extremely stable silica system condensation liquid is obtained can be provided, and the dissolving method of the silica system condensation liquid raw material which a transportation cost is [2nd] low, and economic effectuation is high, and can dissolve a several times the amount raw material can be provided.

[0095]The 3rd can be provided with the manufacturing method of silica system condensation liquid applicable to a actual device, and the 4th can be provided with the manufacturing installation of compact silica system condensation liquid.

[0096]The silica system condensation liquid obtained by this invention is very effective in oily water separation of the oil (animal oil, vegetable oil, straight mineral oil) content wastewater which is used for the adsorption of a dissolved solid, an emulsion substance, a dispersing agent, oil, a suspended solid, etc., the condensation, and exclusion which are contained underwater, and is made difficult especially now. It is thought that such an operation is based on the influence of monomer silica. That is, monomer silica has a function which adsorbs the organic matter of an organic substance, a surface-active agent, or others, inorganic ion, plankton and bacteria, and a suspended solid, or is condensed. This function is not looked at by aluminum system, Fe system, and the polymers system flocculating agent. It is because these are the characteristics which monomer silica has, namely, monomer silica polymerizes, micell is promptly formed between underwater organic matters etc. in the process to gel, and start an interaction between inorganic ion, combine with it and promote the gelling reaction of silica quickly.

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EXAMPLE

[Example]Next, the example of the manufacturing method of the silica system condensation liquid concerning this invention is described based on a drawing.

[0025]Drawing 1 is a manufacture system flow figure of silica system condensation liquid.

[0026]A switch is turned on, a system is started and a constant rate of water is first supplied to a dissolver. This water is not exceptionally limited, although tap water is usually used.

However, the water which contains not less than several PPM for metal ions, such as Fe ion and Al ion, starts silica and an interaction, and is not preferred.

[0027]Next, if the quantity of water becomes fixed, the water in a dissolver will be agitated.

[0028]Next, sulfuric acid is poured in and dilute sulfuric acid of prescribed concentration is created. In the meaning which prevents gelling of monomer silica, as for dilute-sulfuric-acid concentration, less than 2N is preferred, and it is less than 1N more preferably. In this example, it is considered as 1N dilute sulfuric acid.

[0029]As for the sulfuric acid to pour in, when concentrated sulfuric acid is used and acceptance equipment etc. are taken into consideration, accepting by concentration 64% is preferred. It can also be used when existing has concentrated-sulfuric-acid equipment (Fire Service Law full equipment) 98%.

[0030]Next, it holds for several minutes (it is neglected under churning). It is for obtaining 1N uniform dilute sulfuric acid. There should just be about 3 minutes of retention time.

[0031]Next, a silica system condensation liquid raw material is thrown in in a dissolver. As used in this invention, the "amount of solution equilibriums" of a silica system condensation liquid raw material means the quantity of the silica system condensation liquid raw material which can dissolve in the given amount of sulfuric acid. The quantity of the silica system condensation liquid raw material of the degree 1.15 of Ca salt group which can dissolve with 1 l. of 1N dilute sulfuric acid in this example is 42.5g.

[0032]Next, it holds until it reaches a solution equilibrium. Although about 5 minutes may be

sufficient as retention time, by the time it reaches a solution equilibrium more certainly, for 15 minutes is preferred.

[0033]Next, it judges whether buys and there is that pH in a dissolver is over 2.5 as occasion demands. If the reaction of dilute sulfuric acid and a raw material advances, pH in a dissolver will rise. And it holds for several minutes until pH exceeds 2.5, since the reaction is not yet advancing to a solution equilibrium when pH is less than 2.5. By this maintenance, a reaction advances and pH rises. If a reaction is made to continue, in order that pH may converge, if the convergence relation of the reaction time and pH is held, this pH judgment is unnecessary daringly. When attaining low cost-ization in an equipment surface, it is preferred to take the method which does not make this pH judgment.

[0034]On the other hand, since it can judge that the reaction advanced and the solution equilibrium was reached when pH is 2.5 or more, it is judged whether next it filters.

[0035]And it accepts in a condensation fluid tank, including sludge, when not filtering. The silica system condensation liquid of this invention can be obtained by this.

[0036]In filtering, a filter separates into filtrate and sludge, and filtrate is received in a condensation fluid tank. The silica system condensation liquid of this invention can be obtained by this.

[0037]As for the last pH within the dissolver in the case of filtering, it is also preferred to increase and adjust the quantity of a silica system condensation liquid raw material so that it may be set to pH**3.0.

[0038]Sludge is CaSO_4 and $2\text{H}_2\text{O}$ (gypsum fibrosum), and accepts this for example, in a sludge pit.

[0039]When the silica system condensation liquid of this invention in a condensation fluid tank is used, it decreases and it is detected with a level sensor, it returns to a start and the above-mentioned method is repeated again.

[0040]As for the above-mentioned system, automating is preferred, and it clarifies concrete interlocking relationship in the following examples of a device.

[0041]The silica system condensation liquid raw materials used for the manufacturing method of the silica system condensation liquid of this invention are the composite crystal mineral and amorphous mineral of silica.

For example, natural cristobalite, blast furnace slag, etc. are used.

Although silica system condensation liquid raw materials by which reforming treatment was carried out, such as oxidation treatment, i.e., the silica system condensation liquid raw material from which the non-sulfur oxide ingredient of the composite crystal mineral and the amorphous mineral of silica was removed, are specifically preferred, the silica system condensation liquid raw material indicated to Japanese Patent Application No. No. 296371 [one to] can also be used.

[0042]Next, the example of the manufacturing installation of the silica system condensation liquid concerning this invention is described based on drawing 2.

[0043]In drawing 2, 1 is a sulfuric acid injection means, for example, comprises the sulfuric acid tank 101 received as sulfuric acid 64%, the sulfuric acid pump 102, the pipette-like sulfuric acid fixed-quantity container 103, and the sulfuric acid injection valve 104. A corrosion-resistant pump is used for the sulfuric acid pump 102. The pipette-like sulfuric acid fixed-quantity container 103 makes the sulfuric acid sent from the sulfuric acid pump 102 overflow from the overflow pipe 105 in which it was provided by the small tube part of the sulfuric acid fixed-quantity container 103, and is constituted so that a fixed quantity is possible.

[0044]2 is a dissolver and is formed with the product made of resin, or an anticorrosion metal. 201 is an agitator for agitating the liquid in the dissolver 2, and 3 is a tap water injection valve which constitutes a tap water injection means.

[0045]In the dissolver 2, the high-level sensor 202 and the low level sensor 203 are formed. The high-level sensor 202 is being interlocked with the sulfuric acid pump 102, the sulfuric acid injection valve 104, and the agitator 201 respectively, and the low level sensor 203 is being interlocked with the sulfuric acid injection valve 104, the agitator 201, and the tap water injection valve 3 respectively.

[0046]204 is a pH meter formed as occasion demands in the dissolver 2, and is taken as the index which that pH is over 2.5 judges whether buys and there is. However, since a pH value is converged by the reaction time of sulfuric acid and a raw material as mentioned above, a sequence can also be constructed by a timer.

[0047]4 is a means to supply the silica system condensation liquid raw material 401 to the dissolver 2, and is constituted by the feeding feeder 403 formed when carrying out automatic feed to the hopper 402. To not carry out automatic feed, the feeding feeder 403 is unnecessary. In the hopper 402, it may have an air feeding means for oxidizing the silica system condensation liquid raw material 401 within a hopper.

[0048]5 is a condensation fluid tank for receiving silica system condensation liquid.

[0049]pH is 2.5 or more, and the solution obtained within the dissolver 2 is directly sent to the condensation fluid tank 5 with the liquid-sending pump 501, when not filtering. 502 is an agitator and 503 is a valve.

[0050]On the other hand, pH is 2.5 or more, and the solution obtained within the dissolver 2 is sent to the filter 6 with the liquid-sending pump 501, when filtering a solution. In the filter 6, it separates into filtrate and sludge and filtrate is sent to the condensation fluid tank 5. Sludge is stored by the sludge pit 601.

[0051]Various waste water treatment is presented with the silica system condensation liquid in the condensation fluid tank 5 with the treated water pump 7. The treated water pump 7 stops by detection of the low level sensor 504.

[0052]When pH is 2.5 or more and the solution obtained within the above-mentioned dissolver 2 does not filter, various waste water treatment can also be directly presented via the line 505 with the liquid-sending pump 501.

[0053]Next, although the main sequences preferably adopted as the dissolving method of this invention and the manufacturing method of silica system condensation liquid, and a device are explained, it is not necessarily limited.

[0054]Sulfuric acid is received in the sulfuric acid injection means 1 64%, and specified quantity measuring is carried out at the sulfuric acid fixed-quantity container 103. moreover -- opening the tap water injection valve 3 -- the inside of the dissolver 2 -- water -- ** -- a fixed quantity is filled.

[0055]The agitator 210 is started in this state. Subsequently, the sulfuric acid injection valve 104 is opened and the specified quantity is poured in.

[0056]For [time lag] 5 minutes is provided, and if the feeding feeder 403 is started and specified quantity supply is carried out, it will stop.

[0057]For [time lag] 15 minutes is provided, the valve 503 is opened, and the liquid-sending pump 501 is started. Operation of this liquid-sending pump 501 is interlocked with the filter 6 (however, when it needs filtration). Therefore, the filter 6 is also started simultaneously with starting of the liquid-sending pump 501.

[0058]By detection of the low level sensor 203, the liquid-sending pump 501 is suspended, the valve 503 is closed and operation of the filter 6 is suspended.

[0059]Filtrate is received in the condensation fluid tank 5, and the agitator 502 is run continuously at this time.

[0060]If the treated water pump 7 operates, the oil level in the condensation fluid tank 5 falls and the low level sensor 504 detects, the treated water pump 7 will stop, and the tap water injection valve 3 will open, and tap water will be supplied to the dissolver 2. The tap water injection valve 3 is closed by detection of the high-level sensor 202.

[0061]By detection of the low level sensor 203, the tap water injection valve 3 is opened and tap water can be supplied to the dissolver 2.

[0062]

[Example(s) of Experiment]

It experimented using the device shown in example of experiment 1 drawing 2. First, the tap water injection valve 3 was opened to the 150-l. dissolver 2, and 100 l. of tap water was supplied to it.

[0063]Agitating the liquid in this dissolver 2 with the agitator 201, the sulfuric acid injection valve 104 is opened, 4.34 l. of sulfuric acid is poured in 64%, and it was made to become dilute sulfuric acid of 1N concentration.

[0064]Subsequently, the silica system condensation liquid raw material which has the degree

1.15 of Ca salt group was thrown in for 4.25 kg in this dissolver 2.

[0065]Churning was continued and held for 15 minutes and silica system condensation liquid was obtained.

[0066]The viscosity of this silica system condensation liquid was measured as an index, and the polymerization gelling state was investigated. It settled and sol gelling stages of progress were investigated.

[0067]The viscosity meter performed measurement of viscosity.

[0068]A measurement result is shown in Table 1.

[0069]It experimented using the device shown in drawing 2 like the example 1 of example of comparative experiments 1 experiment. First, the tap water injection valve 3 was opened to the 150-l. dissolver 2, and 100 l. of tap water was supplied to it.

[0070]Subsequently, the silica system condensation liquid raw material which has the degree 1.15 of Ca salt group was thrown in for 4.25 kg in the dissolver 2, agitating the liquid in the dissolver 2 with the agitator 201.

[0071]Subsequently, the sulfuric acid injection valve 104 is opened, 4.34 l. of sulfuric acid is poured in 64%, and it was made to become dilute sulfuric acid of 1N concentration.

[0072]Churning was continued and held for 15 minutes and silica system condensation liquid was obtained.

[0073]The viscosity of this silica system condensation liquid was measured as an index, and the polymerization and the gelling state were investigated.

[0074]Measurement of viscosity was made to be the same as that of the example 1 of an experiment.

[0075]A measurement result is shown in Table 1.

[0076]

[Table 1]

	シリカ系凝集液の粘度	ゲル化の進行
実施例 1	1.4 センチポイズ	168 時間後も安定
比較実施例 1	2.3 センチポイズ	24 時間後ゾルゲル化→48 時間後ゲル化

It turns out that the polymerization of silica and gelling are not following the silica system condensation liquid of this invention so that clearly from Table 1. Although sol gelling was accepted 24 hours afterward and gelled 48 hours afterward in the example 1 of comparative experiments, the example 1 of an experiment was stable.

[0077]It experimented using the device shown in example of experiment 2 drawing 2. First, the tap water injection valve 3 was opened to the 150-l. dissolver 2, and 100 l. of tap water was supplied to it.

[0078]Agitating the water in this dissolver 2 with the agitator 201, the sulfuric acid injection valve 104 is opened, 4.34 l. of sulfuric acid is poured in 64%, and it was made to become dilute sulfuric acid of 1N concentration.

[0079]Subsequently, the silica system condensation liquid raw material which has the degree 1.15 of Ca salt group was thrown in for 4.25 kg in this dissolver 2. Stirring was continued for 15 minutes and the silica system condensation liquid of 1N dilute-sulfuric-acid dissolution was obtained.

[0080]4.34 l. was added for sulfuric acid 64%, and the silica system condensation liquid raw material which has the degree 1.15 of Ca salt group in this was thrown in for 4.25 kg.

[0081]Churning was continued and held for 15 minutes and high-concentration silica system condensation liquid was obtained.

[0082]The viscosity of this silica system condensation liquid was measured as an index, the polymerization and the gelling state were investigated and settled, and the stages of progress of sol gelling were investigated.

[0083]Measurement of viscosity was made to be the same as that of the example 1 of an experiment.

[0084]A measurement result is shown in Table 2.

[0085]It experimented using the device shown in drawing 2 like the example 2 of example of comparative experiments 2 experiment. First, the tap water injection valve 3 was opened to the 150-l. dissolver 2, and 100 l. of tap water was supplied to it.

[0086]Agitating the liquid in this dissolver 2 with the agitator 201, the sulfuric acid injection valve 104 is opened, 8.68 l. of sulfuric acid is poured in 64%, and it was made to become dilute sulfuric acid of 2N concentration.

[0087]Subsequently, the silica system condensation liquid raw material which has the degree 1.15 of Ca salt group was thrown in for 8.5 kg in this dissolver 2.

[0088]Churning was continued and held for 15 minutes and high-concentration silica system condensation liquid was obtained.

[0089]The viscosity of this silica system condensation liquid was measured as an index, the polymerization and the gelling state were investigated and settled, and the stages of progress of sol gelling were investigated.

[0090]Measurement of viscosity was made to be the same as that of the example 1 of an experiment.

[0091]A measurement result is shown in Table 2.

[0092]

[Table 2]

	シリカ系凝集液の粘度	ゲル化の進行
実施例 1	1.3 センチポイズ	168 時間後も安定
比較実施例 1	2.1 センチポイズ	24 時間後ゾルゲル化→48 時間後ゲル化

The silica system condensation liquid of this invention is understood that the content of monomer silica is clearly high in spite of being high concentration, and the stability of monomer silica is high so that clearly from Table 2.

[0093] That there are few monomer silica contents of comparative silica system condensation liquid, and the stability of monomer silica is missing, In the place where pH rises selectively by addition of a silica system condensation liquid raw material, polymerization-ization of silica takes place and it is thought in the low place of pH that it originates in anhydrous silica being generated. If the polymerization of silica and sol gelling are accepted, gelling will advance quickly and it will become a jelly state solid.

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[Translation done.]

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TECHNICAL FIELD

[Industrial Application]The dissolved solid, the emulsion substance, the dispersing agent, oil which contain this invention underwater, A dissolving method of the silica system condensation liquid raw material used as the raw material for manufacturing the silica system condensation liquid used for adsorption of a suspended solid etc., condensation, and exclusion, It is related with the dissolving method of the silica system condensation liquid raw material for obtaining detailed silica system condensation liquid with a high content ratio of monomer silica, the manufacturing method of silica system condensation liquid, and the manufacturing installation of the silica system condensation liquid in which a compact design is possible about the manufacturing method and device of silica system condensation liquid.

[0002]

[Background of the Invention]Conventionally, aluminum system condensation liquid, Fe system condensation liquid, and polymers system condensation liquid are mainly used for extraction of the dissolved solid (ion) in sewage or various waste water, organic and inorganic particles, a suspended solid, etc., etc., or condensation and removal.

[0003]Since the function which this conventional aluminum system and Fe system condensation liquid adsorb an organic chemistry substance, oil, an oil emulsion, a surface-active agent, etc., or it condenses is very weak, it faces actually using it, Precipitation of a lot of aluminium hydroxide and iron hydroxide is made to generate using a lot of aluminum systems and Fe system condensation liquid (also adding alkali further), Oil etc. are physically hooked on those settlings, and also a polymer coagulant is prescribed for the patient, flocks shape is enlarged, and floatation or the way sedimentation separates flocks with water is taken.

[0004]Even if it performs this complicated processing, it is difficult to obtain a satisfactory result, and also a lot of sludge resulting from the flocculating agent prescribed for the patient occurs, and there is a problem which must also pay the cleanup cost collectively.

[0005]In order to solve this problem, silica system condensation liquid is proposed in the

Japanese-Patent-Application-No. No. 296371 [one to] specification. The profits that the sludge of this silica system condensation liquid which adsorbs effectively those organic chemistry substances, oil, an oil emulsion, a surface-active agent, etc., condenses them, and is generated also decreases in a degree very much are obtained.

[0006]Although obtained by conventional silica system condensation liquid dissolving a silica system condensation liquid raw material in neutral acid or the reductic acid, and a concrete target at dilute sulfuric acid, When dissolving a silica system condensation liquid raw material, after dissolving a silica system condensation liquid raw material in water, by the method of adding dilute sulfuric acid, it turned out that a silica system condensation liquid raw material becomes sol gel, and there is a fault which lacks in stability few in the rate of monomer silica. Although the statement of the purport that a silica system condensation liquid raw material is added to dilute sulfuric acid is accepted in conventional technology, recognition of the purport that an addition order influences the rate of monomer silica greatly does not exist at all.

[0007]Although the silica system condensation liquid which dissolved double the amount of the raw material from the efficiency of transportation, etc. was called for, it turned out that the monomer silica needed depending on a manufacturing method decreases. Namely, if a silica system condensation liquid raw material is dissolved in 1 l. of dilute sulfuric acid of 1N by the dissolution equivalent, the silica system condensation liquid of 1N will be obtained, but. If the dissolution equivalent part (twice at time of 1N as many quantity as this) dissolution of the silica system condensation liquid raw material is carried out, to 1 l. of dilute sulfuric acid of 2N, noting that I will obtain the silica system condensation liquid of 2N like this, In the place where pH rises selectively by addition of a silica system condensation liquid raw material, polymerization-ization of silica took place and it turned out in the low place of pH that anhydrous silica is generated and the monomer silica needed decreases.

[0008]furthermore -- needing at least 2 liquid, when using conventional aluminum system condensation liquid, Fe system condensation liquid, and polymers system condensation liquid -- so much -- also coming out -- the number of devices increased and miniaturization of the device was made difficult. In the art of Japanese Patent Application No. No. 296371 [one to], the concrete manufacturing method and device of silica system condensation liquid were not clear, and development of a manufacturing method and a device applicable to a actual device was desired.

[Translation done.]

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CLAIMS

[Claim(s)]

[Claim 1]Supplying a constant rate of water to a dissolver, and agitating water in this dissolver, when dissolving a silica system condensation liquid raw material with dilute sulfuric acid and manufacturing silica system condensation liquid. A dissolving method of a silica system condensation liquid raw material supplying the amount of solution equilibriums which can dissolve a silica system condensation liquid raw material which has the specific degree of Ca salt group with said dilute sulfuric acid in this dissolver after pouring in sulfuric acid and creating dilute sulfuric acid of prescribed concentration.

[Claim 2]In a dissolving method of a silica system condensation liquid raw material in which a content ratio of monomer silica is high, and high concentration silica system condensation liquid is obtained, A dissolving method of a silica system condensation liquid raw material obtaining silica system condensation liquid by a method according to claim 1, adding sulfuric acid by the same equivalent as said dilute sulfuric acid subsequently, supplying the amount of solution equilibriums which can dissolve said silica system condensation liquid raw material with this dilute sulfuric acid again, and repeating this operation as occasion demands.

[Claim 3]In a manufacturing method of silica system condensation liquid which dissolves a silica system condensation liquid raw material which has the specific degree of Ca salt group with dilute sulfuric acid, and manufactures silica system condensation liquid, After pouring in sulfuric acid and creating dilute sulfuric acid of prescribed concentration, supplying a constant rate of water to a dissolver, and agitating water in this dissolver, After holding until it throws in a silica system condensation liquid raw material and reaches subsequently to a solution equilibrium in this dissolver, Next, it judges whether buys and there is that pH in this dissolver is over 2.5 as occasion demands, It holds for several minutes until pH exceeds 2.5, when this pH is less than 2.5, A manufacturing method of silica system condensation liquid using filtrate as silica system condensation liquid in judging whether it next filters on the other hand when

pH is 2.5 or more, considering it as silica system condensation liquid as it is in not filtering, and filtering.

[Claim 4]A manufacturing installation of silica system condensation liquid characterized by comprising the following.

A dissolver.

A water supplying means which supplies a constant rate of water to this dissolver.

An agitating means which agitates liquid in this dissolver.

A sulfuric acid injection means for pouring sulfuric acid into a dissolver to which water was supplied from said water supplying means, a means to supply a silica system condensation liquid raw material to this dissolver, and a condensation fluid tank for receiving silica system condensation liquid, when pH in this dissolver is 2.5 or more and it does not filter a solution in this dissolver.

[Claim 5]A manufacturing installation of silica system condensation liquid characterized by comprising the following.

A dissolver.

A water supplying means which supplies a constant rate of water to this dissolver.

An agitating means which agitates liquid in this dissolver.

A condensation fluid tank for receiving silica system condensation liquid which a sulfuric acid injection means for pouring sulfuric acid into a dissolver to which water was supplied from said water supplying means, a means to supply a silica system condensation liquid raw material to this dissolver, and pH in this dissolver are 2.5 or more, and is this filtrate when filtering a solution in this dissolver.

[Translation done.]